LASER ASSISTED TITANIUM CARBIDE (TiC) COATING ON AISI304 STAINLESS STEEL

A Thesis Submitted in Partial Fulfillment of the Requirements for the Award of the Degree of
Master of Technology
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Mechanical Engineering
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By

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DECLARATION

I the undersigned solemnly declare that the report of work entitled "***Laser Assisted Titanium Carbide (TiC) Coating on AISI304 Stainless Steel"*** is based on my own work carried out during the course of my study under the supervision of Dr. M. Masanta.

I assert that the statements made and conclusions drawn are an outcome of the project work. I further declare that to the best of my knowledge and belief that the report does not contain any part of any work which has been already submitted for thesis evaluation in this University.

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Certificate

This is to certify that the report entitled " ***Laser Assisted Titanium Carbide (TiC) Coating on AISI304 Stainless Steel"*** submitted by ***Ms. K. Ushashri*** to National Institute of Technology Rourkela, is a record of bonafide research work carried out by her under my supervision and is worthy of consideration for thesis evaluation in Mechanical Engineering with specialization in **Production.** The embodiment of this report has not been submitted in any other University and/or Institute for the award of the any degree or diploma.

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K .Ushashri

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ABSTRACT

The increasing interest in the betterment of wear resistance and hardness of surfaces that are in contact with abrasives or corrosive materials has accelerated the development of several techniques for creating protective coatings. Laser coating is one such advantageous process of modifying the surface properties of different tools, parts of long running machines, etc. In this project a search is done on what can be the possible coatings (ceramic particles embedded in metal matrix) and which are the substrates over which this coating is feasible. TiC was found to be exhibiting a very high melting point and thermal stability, high hardness and excellent wear resistance, low coefficient of friction and high electrical and thermal conductivities and hence chosen to be the best coating available. The substrate that is chosen for coating is AISI304 stainless steel. For the laser treatment pulsed Nd-YAG laser is used. After the laser coating operation the phase changes was studied under SEM. Composition of the laser clad layers was obtained by XRD and the hardness was measured by using Vickers microhardness tester**.** The hardness of the coating increased to a substantial amount in the range of 650-1900 $HV_{0.1}$ as compared to 190 $HV_{0.1}$ hardness of steel substrate. The coating thickness, the extent of dilution of the stainless steel substrate by TiC and the hardness were primarily dependent upon the laser parameters of power, frequency and the scanning speed. From the microstructure analysis it is revealed that at low values of frequency and peak power a thick coating of TiC is more uniformly distributed over the laser treated tracks and at higher values of frequency and peak powers a MMC type of coating is obtained on the substrate. From the analysis it is concluded that increasing the peak power, the intensity of Fe on the surface increases and with increasing frequency the intensity of TiC phase on the surface decreases keeping the scan speed constant. Hardness values of the coating decreases with increasing value of laser pulse frequency when peak power is constant and also decreasing with increasing laser peak power when laser pulse frequency is constant.

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List of abbreviations

Al	: Aluminium
Al_2O_3	: Aluminium oxide
C	: Carbon
CO2	: Carbon dioxide
Cr	: Chromium
CrB	: Chromium boride
$Cr_{23}C_6$	: Chromium carbide
CNC	: Computer numerical control
CrO	: Chromium oxide
Co	: Cobalt
CVD	: Chemical vapour deposition
Fe	: Iron
F_{freq}	: Frequency of pulse
HV	: Vickers pyramid number
LSE	: Laser surface engineering
MMC	: Metal matrix composites
Mg	: Magnesium
Mn	: Manganese
Mo	: Molybdenum
Nd-YAG	: Neodymium-doped yttrium aluminium garnet
Ni	: Nickel
P	: Phosphor
P_{avg}	: Average power
P_{peak}	: Peak power
PVD	: Physical vapour deposition
S	: Sulphur
SEM	: Scanning electron microscope
Si	: Silicon
SiC	: Silicon carbide
Ti	: Titanium
TiB_2	: Titanium boride

TiC	: Titanium carbide
T_{on}	: Time for which laser is on per pulse
VHN	: Vickers hardness number
WC	: Titanium carbide
XRD	: X-ray diffractor
ZrO_2	: Zirconium oxide
n	: Order of the diffracted beam
λ	: Wavelength of the incident X-ray beam
d	: Distance between adjacent planes of atoms (the *d*-spacing)
θ	: Angle of incidence of the X-ray beam

CHAPTER-1

INTRODUCTION

- Different techniques used for surface coating
- Different types of laser surface treatments
- Laser surface treatment in which external material is used: laser alloying, dispersing and cladding
- Different type of lasers that are used for laser surface treatments
- Laser coating methods
- Energy redistribution in laser coating process
- Applications of laser surface coating technique
- Laser coating layer properties
- Process parameters that affect the coating layer properties

1. Introduction

Laser coating is a process in a substrate is fused, with a material (coating powder) which has metallurgical properties that are different from substrate, by using a laser beam. The peculiarity of the process is that only a very thin layer of the substrate is to be melted in order to achieve metallurgical bonding. In order to maintain the original properties of the coating materials, added material and substrate should have minimum dilution. Thus it is an advanced coating technology for improving the properties of various components. Coatings so obtained by laser treatment have characteristics of extremely high density, non porous and crack free microstructure and excellent metallurgical bonding with the base material.

1.1 Different techniques used for surface coating

There are different surface engineering techniques used to improve the surface properties that are used in the present days. Among these are physical vapour deposition (PVD), chemical vapour deposition (CVD), plasma spraying, electro-deposition, carburizing, nitriding, flame/induction hardening, galvanizing, diffusion coating, etc. Some of them are explained below:

- Physical vapour deposition (PVD):
  It is a process to produce metal vapour that can be deposited on electrically conductive materials as a thin highly adhered pure metal or alloy coating.

- Chemical vapour deposition:
  It is a process in which thin-film coatings are formed as a result of reactions between various gaseous phases and the heated surface of substrates within the CVD reactor. As different gases are transported through the reactor, distinct coating layers are formed on the tooling substrate. For example, TiN is formed as a result of the following chemical reaction:

$$TiCl_4 + N_2 + H_2 \rightarrow TiN + 4\ HCl + H_2$$

- Thermal / Plasma spraying:
  It is basically the spraying of molten or heat softened material onto a surface to provide a coating. Material in the form of powder is injected into a very high temperature

plasma flame, where it is rapidly heated and accelerated to a high velocity. The hot material impacts on the substrate surface and rapidly cools forming a coating.

- Electrodepositing:
  It is a sophisticated form of dip coating that involves immersing a conductive part into a conductive waterborne coating solution. Electro deposition coating is also called electro coating and electrophoretic coating.

- Carburizing:
  It is a process of adding Carbon to the surface.

- Nitriding:
  It is a process of diffusing Nitrogen into the surface of steel.

However, these techniques offer many limitations, e.g. they require high processing time, high input energy, consumption of material is high, lack of flexibility, poor precision, and lack in scope for automation. On the other hand laser surface engineering techniques (e.g. laser cladding or laser alloying) which are based on applications of laser beam are free from many of these limitations.

Laser assisted surface engineering offers several advantages over other surface modification techniques.

I. The total heat input from laser is low i.e. the heat affected zone is small, resulting in minimal distortion and thus very local treatment without affecting the bulk substrate is possible.
II. The processing time is very less and it can be used on materials which are hard and are having high melting points.
III. Complex support systems like ultra-high vacuum environment etc are not required by the process as the processing is least affected by the environment.
IV. Laser surface modification is a non-equilibrium synthesis.

V. Minimal dilution of the substrate by the coating material is possible however producing a sound and adherent interface is obtained between the coating and substrate which are metallurgically bonded.

VI. The surface microstructure can be controlled by altering process variables such as laser power, scan speed, beam diameter and coating powder composition.

VII. In laser cladding the energy supplied can be well controlled and the process depth is very well defined beforehand.

Thermal spraying and vapour deposition (physical and chemical) also produce hard, wear resistant and corrosion resistant coatings but the three surface engineering techniques, thermal spraying, PVD/ CVD and laser cladding differ in the ways given in the Table 1.

Table 1: Differences between different surface coating techniques

Surface engineering technique → / Basis of difference ↓	Thermal spraying	PVD/CVD	Laser cladding
Heat source	Combustion flame, electric or plasma arc	High power electric arc or electron bombardment(in case of PVD) and hot filament (in case of CVD)	High intensity laser radiation
Working environment	Atmospheric conditions	Vacuum (PVD) /reactive hydrogen gas atmosphere (CVD)	Normal atmospheric conditions
Deposition rates	Low (in case flame spraying) and high (in case of wire flame)	Low	High
Dilution	Nil	Nil	Low
Coating thickness	Thick coatings (25 µm to 2.5 mm)	5 to 12 µm	0.5-3mm

1.2 Different types of laser surface treatments

Initially we saw different types of surface engineering techniques. Among these we found that laser surface engineering technique was the preferred one. Again the laser surface treatment can be classified as follows:

- Laser treatment without use of external material
    - I. Laser transformation hardening
    - II. Laser melting
- Laser treatment with use of external material
    - I. Laser alloying
    - II. Laser dispersing
    - III. Laser cladding

      Laser cladding is again done by two methods:
        - i. By powder feeding method
        - ii. By preplaced powder method

Figure 1shows the two classifications of laser surface treatments.

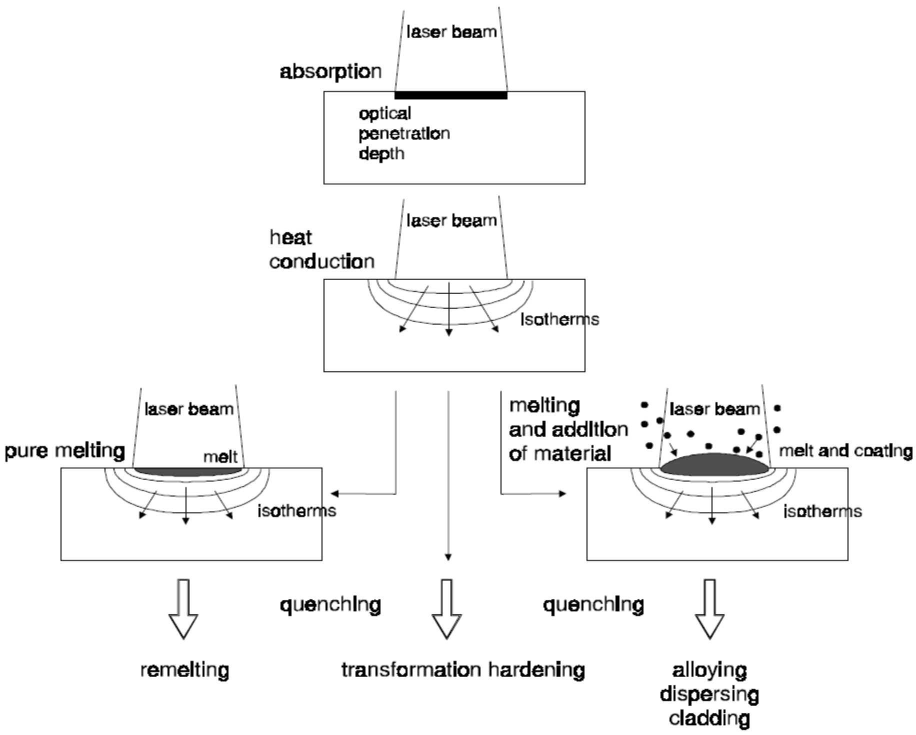


Fig.1: Laser surface treatments are distinguished with respect to the surface temperature (solid phase/melt pool) and to the addition of material. (Ref- Schneider M.F, 1998 [39])

1.3 Laser surface treatment in which external material is used: laser alloying, dispersing and cladding

The three techniques, Laser alloying, laser dispersing and laser cladding involve the formation of a melt pool of coating and substrate material. But they can be distinguished on the basis of degree of mixing between the coating material and the base or substrate material in the surface layer.

Laser alloying involves complete mixing and reaction between added elements and the base material in the surface region. On the contrast laser cladding generates a surface layer that hardly contains elements of the substrate, but enough mixing is allowed to achieve a strong bonding at the interface of laser clad and the substrate. Hence the properties of the produced clad layer entirely depend on the applied coating material.

Figure 2 shows the degree of mixing of coating material and the substrate in each of the cases.

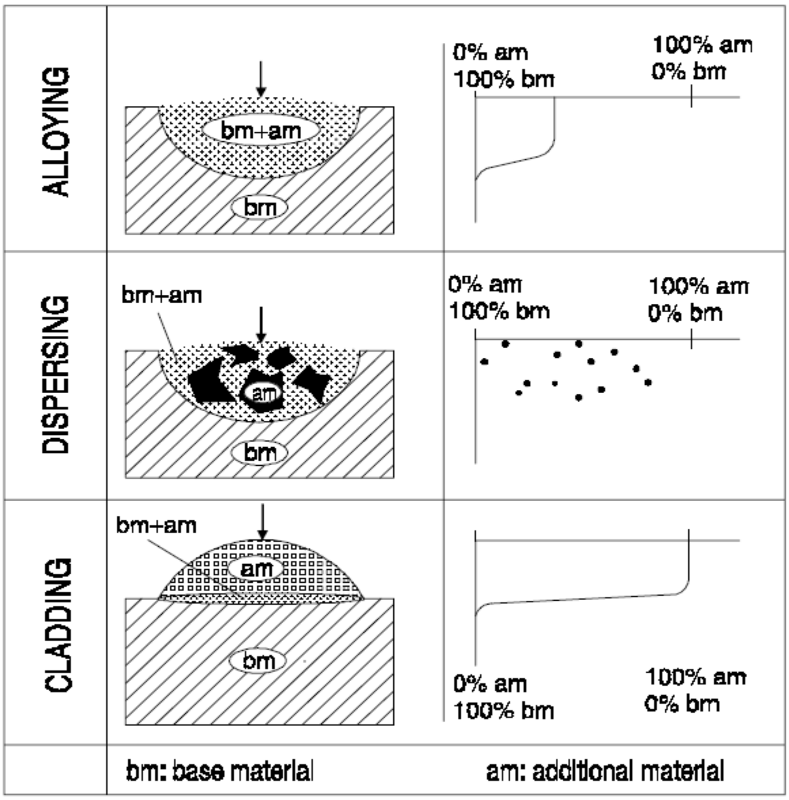


Fig .2: Laser alloying, dispersing and cladding. The right part of the picture indicates the distribution of the added elements measured top-down in the centre of the track. (Ref- Schneider M.F, 1998 [39])

In case of alloying we can see that the base material and additional material have been homogeneously mixed and thus the laser treated surface so produced shows properties of both the coating and substrate material. In dispersing as the name suggests the additional material has been dispersed non-homogeneously in the matrix of base and additional material. Lastly in the laser cladding the laser clad consists of just the additional material.

1.4 Different type of lasers that are used for laser surface treatments

There are many different types of lasers. The laser medium can be a solid, gas, liquid or semiconductor. Lasers are commonly designated by the type of lasing material employed:

a. Solid-state lasers:

These are the lasers having lasing material distributed in a solid matrix (such as the ruby or neodymium: yttrium-aluminium garnet "YAG" lasers). Solid state laser operate at a lower wavelength which improves absorption characteristics i.e. metal surface absorbs better energy from laser beam. Nd: YAG laser cladding process is twice as energy efficient as the CO_2 laser cladding process

b. Gas lasers:

Helium and helium-neon are the most common gas lasers. These lasers have a primary output of visible red light. CO_2 lasers are most traditional high power lasers. They have characteristics of very high power and power density, moderate efficiency, reliable operation and excellent beam quality. They have high wavelength 10.6 μm which results in lower absorption of laser beams by metals.

c. Diode lasers:

With the advent of diode laser, High power diode lasers (HPDL) in the kilowatt range and with larger rectangular beam profiles (almost uniform intensity distribution) become appropriate tools for laser surface engineering. In addition, due to high optical efficiency, and low running costs diode laser become relatively less expensive compared to CO_2 and Nd: YAG lasers. The wavelength of the emitted radiation in case of diode laser allows higher absorption by metallic surfaces than CO_2 laser.

d. Fibre Laser:

In these lasers the active gain medium is an optical fibre doped with rare earth materials like erbium, yttrium, neodymium, thulium etc. delivery of the beam doesn't require any complicated or sensitive optics. Beam quality is very high with very high power generation

1.5 Laser coating methods

Depending on the way the coating powder is added to the substrate surface before or during the laser processing, the laser coating technique is classified as given below which is illustrated in fig 3.

- Pre-deposition laser coating
- Blown powder laser coating

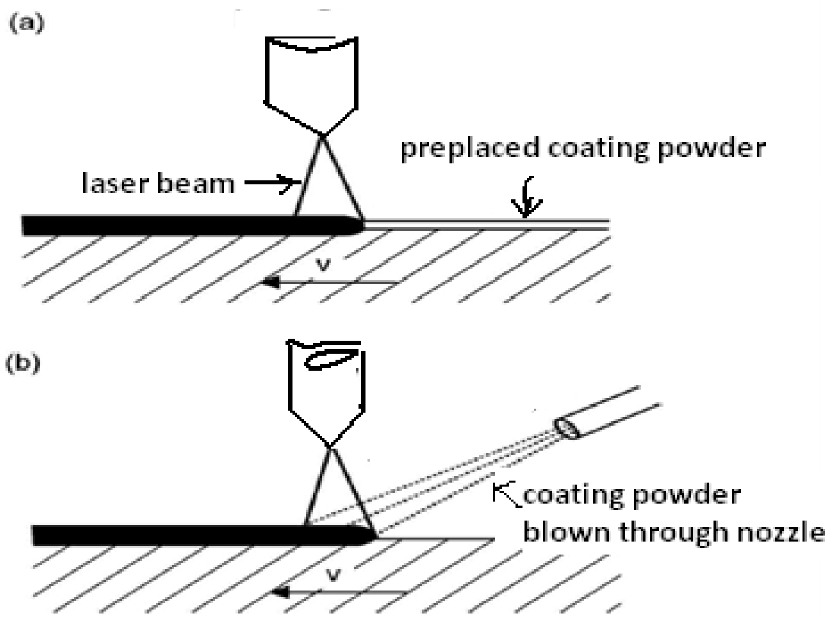


Fig.3: Laser coating by (a) Preplaced powder method and (b) Blown powder method [Ref-H. Gedda, 2004 [38]]

Laser coating with preplaced powder:

Preplaced powder method is the simplest method of the two mentioned above provided that the powder can be made to remain in place until melted. The powder is thus mixed with a binder. The working area is shrouded by an inert gas. The preplaced powder method involves scanning of the laser beam over the powder bed. Figure 4 depicts the preplaced technique.

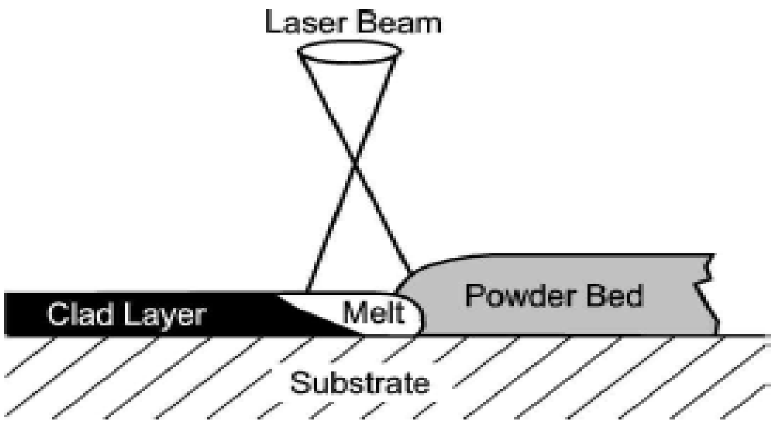


Fig .4: The preplaced powder cladding technique

Preplaced powder coating technique is a three stage melting process. Its three stages are as follows:

I. The laser beam rapidly melts the coating powder before the melt touches the substrate. This is because, prior to contact with substrate, the melt is surrounded by powder which has low conductivity.

II. As the melt touches the substrate it loses a maximum amount of heat by conduction which leads to partial solidification of the melt. As a result the melt liquid interface does not move into the body of the substrate.

III. If the laser energy continues to irradiate from the top surface of the melt, the energy will gradually move the melt/solid interface back through the clad layer and across into the body of the substrate.

Advantages of preplaced powder coating:

- It is cost effective
- Its procedure is simple
- It can be used for testing purposes for small scale production of coated materials

Blown powder method of laser cladding:

Blown powder method as depicted in fig 5 produces a high quality coating layer with minimum dilution. In this method powder is injected into the melt pool by using a carrier gas and the nozzle is directed at an angle of 38^0 to 45^0 (from the base) towards the substrate. The powder particles get heated when they pass through the laser beam. At the interface melting starts and the molten coating powder particles are trapped in the melt pool.

The basic system of blown powder laser coating set up is made up of

- Laser device which generates the beam of optics and direct it
- a powder feeder
- and a part manipulator

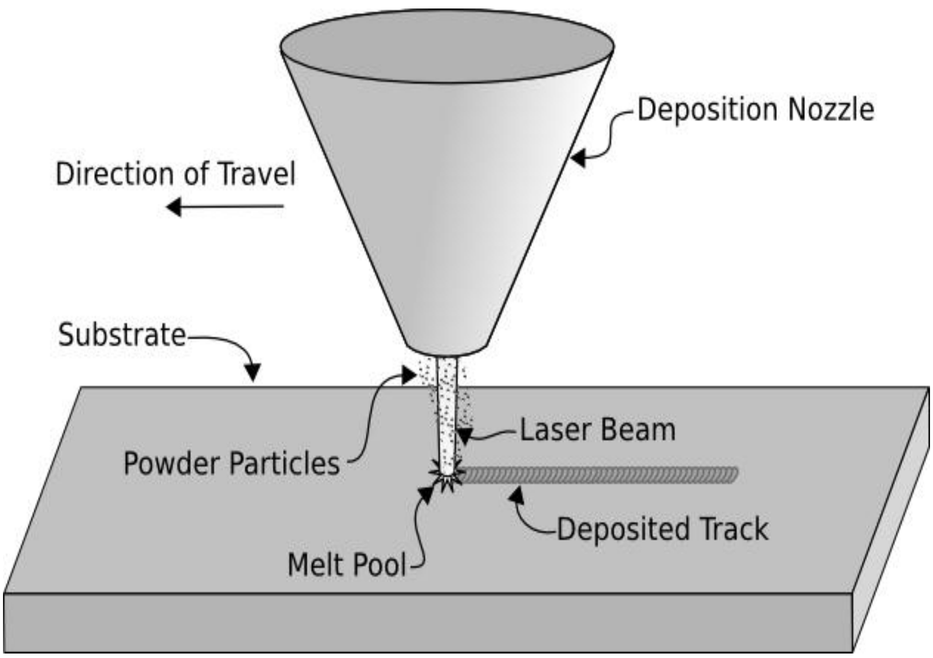


Fig .5: Schematic diagram of blown powder laser coating process (google search)

1.6 Energy redistribution in laser coating process

It is important to analyse the percentage of the laser power that is utilized in the laser coating process and the fraction of it that is reflected, absorbed and radiated. The main energy loss in the process of laser treatment is by reflection from the melt pool and the powder cloud.

The laser power required in the laser surface treatment is redistributed in following ways.

Laser reflected off the coating melt	40% (approx)
Power reflected off the powder cloud	10% (approx)
Power used to heat the substrate	30% (approx)
Power used to melt the clad layer	20% (approx)

Since a large proportion (i.e.30%) of the laser power is consumed in heating of the substrate, it would be profitable if the preheating of the substrate is done by a cheaper power source (flame, plasma etc).

1.7 Applications of laser surface coating technique

I. It is the best technique for coating any shape
II. Ideal for repair and distortion: preferably used for restoring worn blade tips and labyrinth seals (particular depositions for repairing parts).
III. Most suited technique for graded material application.
IV. Optimal part design by dissimilar metal deposition.
V. Material research and development.
VI. Wear resistance and fatigue life improvement.

Laser coating technique is used successfully in various industries:

- Oil drilling- Parts of off-shore drilling heads, Drainage plough blade
- Steel industries - Deep drawing tool, Extruder screw plastic machinery, Moulding die
- Automobile industry- Exhaust valves large diesel engines, Jet engine turbine blade notch, Cylinder and valve, Jet engine turbine blade notch
- Power plants- High pressure gas turbine blade shroud, Leading edge steam turbine blade, Gas turbine airfoil thermal barrier, Compressor blade, Nuclear valve

1.8 Laser coating layer properties

Some of the important properties and characteristics of the laser coating produced by the preplaced powder over the substrate are mentioned in the Table 2.

Table 2: Major properties of laser coating (Ref- Schneider M.F, 1998 [39])

Geometrical properties	**Mechanical properties**	**Metallurgical properties**	**Qualitative properties**
coating height dilution roughness	hardness residual stress wear resistance tensile strength	microstructure dilution grain size homogeneity corrosion resistance	porosity cracking

The laser coating properties are described below:

1. Coating height:

   With increase in feed rate laser coating height increases above the substrate surface and the melts depth within the substrate decreases. In contrast with increase in laser power coating height decreases and melts depth increases.

2. Dilution of laser coating:

   Spreading of melt pool on the substrate takes place after the premixed clad powder over the substrate is exposed to laser. Laser clad layers properties are affected by dilution. Dilution determines the strength of the coating layer which determines the wear resistance of the surface so produced. It is found that increasing the area of dilution for specific limit, the bond strength between clad layer and substrate will be increased. Whereas applications that require good corrosion or oxidation resistance, in them clad layer composition must have maximum percentage of coating material.

3. <u>Roughness</u>:

Roughness signifies lack of surface finish. Thus minimizing the roughness improves the surface quality.

4. <u>Hardness</u>:

The hardness of the coating layer is influenced by the type of coating material and the process speed.

If the coating speed is low, then the amount of heat input is higher during the process and the powder particles get better dissolved into the mass of the surface layer. And thus in this case strengthening effect is more. Microhardness increases with increase in feed rate, increase in laser beam diameter, quantity of coating material and decreases with increase in laser power.

5. <u>Wear resistance</u>:

Wear resistance determines the life of the coated substrate. It depends on the ceramic material used for coating over the substrate and the laser treatment parameters.

6. <u>Crack formation</u>**:**

Cracking is caused due to fast cooling rates and residual stresses. Crack prevention is important, because cracks in the coating surface initiate corrosion fracture and reduce fatigue strength.

7. <u>Residual stress</u>:

During heating the area of substrate which is exposed to laser gets heated and expands. However, it is constrained by the cold surrounding substrate area and becomes stressed in compression until melting occurs that relaxes the stresses. It is during the re-solidification that the tensile stresses are formed due to shrinkage of the melt pool which is limited by the metallurgical bonding with the substrate. And thus the high thermal gradients involved during the process generate residual stresses. Clearly, these residual stresses affect the mechanical properties, such as fatigue, creep and brittle fracture behaviour of the surface formed by coating and the substrate.

8. Homogeneity:

A careful selection of laser processing parameters results in coating layers which has a homogeneous chemical composition that is free from defects.

9. Porosity:

Presence of holes in the coated layer is being referred to as porosity. It is usually as a result of the formation of gas bubbles that are trapped in the solidifying melt pool.

1.9 Process parameters that affect the coating layer properties

Hierarchy diagrams show the different process parameters like laser beam properties, machine parameters and powder properties that affect the laser coating characteristics.

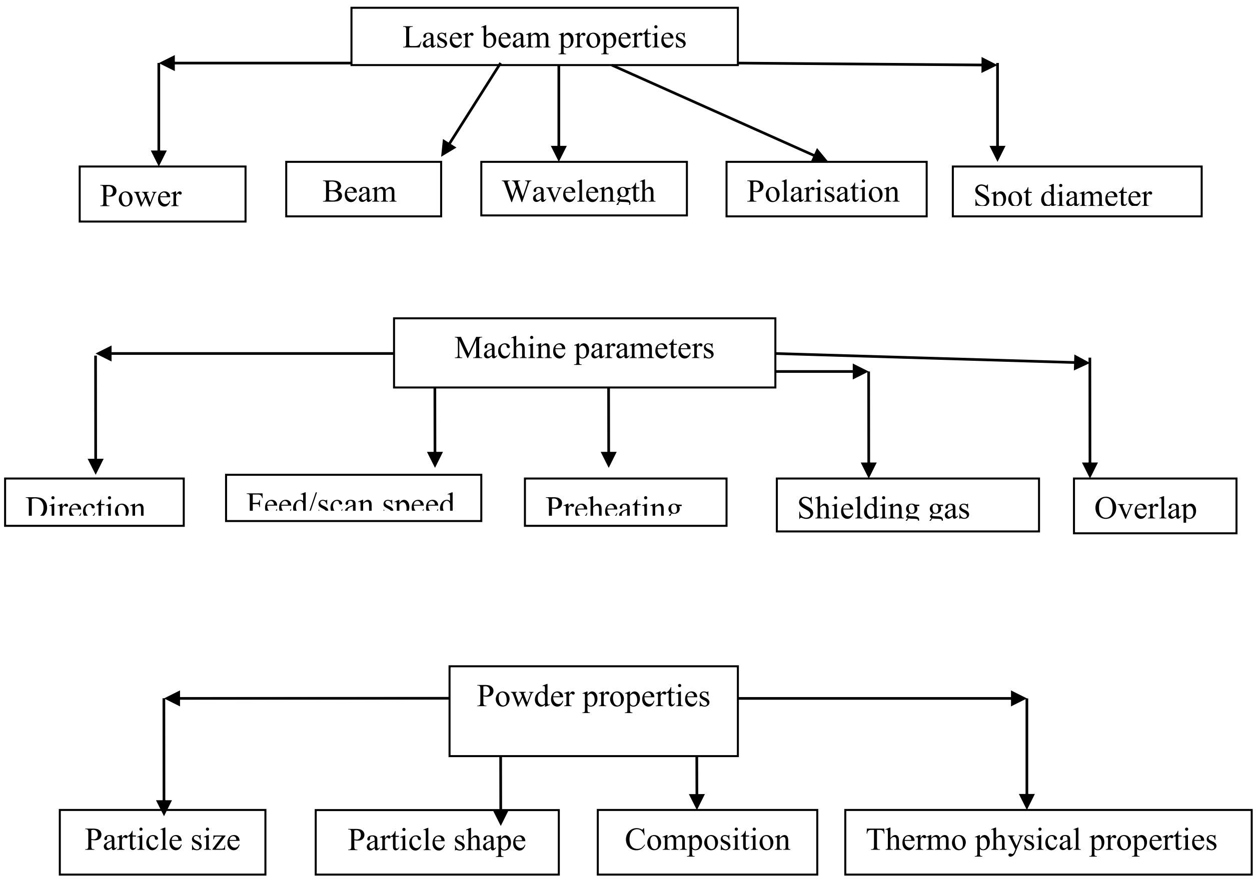


Fig .6: Hierarchy diagrams summarizing the properties which affect the laser coating characteristics

CHAPTER-2

LITERATURE REVIEW

- Metal Matrix Composites
- Laser cladding process
- Laser cladding on AISI304 Stainless steel
- Different research works to improve surface properties of materials

2. Literature review

2.1 Metal Matrix Composites

Recently with development of metal matrix composites (MMC), high performance depositing composite coatings on low grade substrate materials has become an interesting area of research. Particularly, various carbide reinforced MMC coatings have been developed to modify wear and corrosion resistance [1-4]. Metal–matrix composites (MMCs) reinforced with hard ceramic particles have received considerable interest because they can offer improved strength, stiffness and wear resistance compared to their monolithic counterparts. However, a poor toughness of metal matrix composite imposes a serious restriction on fabrication of the bulk materials for structural application [5]. On the other hand, wear is a surface-dependent degradation that may be improved by a suitable modification of the micro-structure and/or composition of the near surface region. Hence, instead of the bulk reinforcement, if a composite layer is developed on the near surface region it would enhance the wear resistance property significantly without affecting the toughness [6].

2.2 Laser cladding process

One such technique to improve the surface properties is laser cladding. Laser composite surfacing is a process where a high-power laser beam is used as a source of heat to melt the metallic substrate and simultaneous feeding of the ceramic particles (in the form of powder) in the molten surface. This is the fabrication of a surface layer of particulate (ceramics like WC, TiC, SiC, ZrO_2 or Al_2O_3) reinforced metal matrix (any of the feasible metal like Ni, Fe, etc) composites on metallic materials [7-12]. Ability to deliver a large power/energy density (10^3–10^5 W/cm^2), high heating/cooling rate (10^3–10^5 K/s) and solidification velocities (1–30 m/s) are the notable advantages associated with laser-assisted composite surfacing [13-15]. This leads to development of wide variety of micro-structures with novel properties that cannot be achieved by conventional processing technique that are more traditional and involve the deposition of films and coatings from solid, liquids and vapour sources [16]. Moreover, these coatings are metallurgically bonded providing a sound and adherent interface between the coating and substrate.

Laser cladding is a process that has brought tremendous interest in a number of industries, as the process can be used to enhance material surface properties for corrosion and wear resistance, repair of worn parts, and forming of near net-shaped structures, as well as customisation of material and mechanical properties through functionally graded materials [17].

2.3 Laser cladding on AISI304 Stainless steel

AISI304 stainless steel possesses the unique combinations of superior mechanical properties and aqueous corrosion resistance and hence, extensively used as structural materials [18]. But due to its low hardness (200 HV), its tribological properties are very poor [19]. The addition of carbon can increase the hardness of stainless steel simultaneously decreasing its ductility. Also heat treatment of stainless steel for increasing its hardenability is not profitable because, heat treatment causes the carbon to combine with the Cr in steel to form chromium carbide which has the formula $Cr_{23}C_6$. This compound forms along the grain boundaries and robs the regions along the grain boundaries of Cr [20].

Cheng et al. studied the dispersion of ceramic particles (WC, CrC, SiC, TiC, CrB and CrO) on austenitic stainless steel UNS S31603 using a laser surfacing technique. The powders were pasted on the surface of UNS S31603 first, followed by surface melting using a high-power laser [21].

2.4 Different research works to improve surface properties of materials

The micro-hardness of the surface was improved to 250-350 VHN as compared to 220 VHN of the AISI 304 stainless steel substrate when it is laser treated with TiB_2 as coating and observed a significant improvement in wear resistance property. The mechanism of wear was found to be a combination of adhesive and abrasive in as-received stainless steel. However, it was predominantly abrasive for laser composite surfaced stainless steel [22].

Direct laser cladding of SiC dispersed AISI304 stainless steel produced defect free and homogeneous microstructure which consists of partially dissolved SiC in grain refined austenite. The hardness increased from 155VHN to 250-340 VHN [23].

TiC exhibits a very high melting point and thermal stability, high hardness and excellent wear resistance, low coefficient of friction and high electrical and thermal conductivities. Because of its high melting point, TiC is a promising material to be used as first wall material in fusion reactors [24].

Hard TiC ceramics are well known for combining a number of special properties that have made them of particular interest for a wide variety of applications-they are used as wear resistant coating for cutting tools and inserts and as diffusion barriers in semi-conductor technology[25].

Different experiments have been conducted with positive results of increased hardness and improved wear and corrosion resistances of different structural materials.

By using a mixture of 80 wt. % 431-type stainless steel and 20 wt. % titanium carbide powders which undergo laser treatment at power of 2430W, a microstructure exhibiting a homogeneous distribution of fine carbide particles was formed, and a 65% increase (724 HV) in hardness compared to a deposit made with only 431 stainless steel powders (438 HV) was observed [26].

Clad hardness is 4 times greater than substrate AISI 1030 medium carbon steel when it is treated under laser coating process with coating as a mixture of Ti (max size of 0.04 mm, 99.5% purity), graphite (max size of 0.04 mm, 99.5% purity) and iron (max size of 0.04 mm, 98% purity) powder [27].

On the substrate of AISI 4140 steel it can be seen that the hardness of the laser coating layers of TiC(reinforcement)/H13 tool steel(matrix) is up to 600–860HV, much higher than that of the substrate(200–250HV), the power being used is 380W [28].

The coating of mixture of three powders TiC, WC and Co (wt%—30:64:6) on 45 steel produce a laser clad of high microhardness, 1000$HV_{0.2}$ [29].

A powder mixture of Ni alloy, titanium (99.7% purity) and crystalline graphite (99.5% purity) was used as the coating alloy on substrate of 5CrMnMo steel which increased its hardness to 1250 $HV_{0.2}$ [30]

Laser coating of 30 vol.% TiC particulates and 70 vol.% Ni-alloy powders on 1045 steel treated under a power of 1000W produces a clad of hardness $HV_{0.2}$=1300 [31].

The values of microhardness of the laser clad layers with different pre-mixed compositions of Mo/TiC are 5–10 times higher than that of the as-received Al alloy. Compared with the as-received Al alloy AA6061, the cumulative wear loss of the laser clad specimens is 20–28 times better [32].

TiC has been deposited on 6061 Al alloy using the LSE technique with laser beam power of 1.8 KW to form a coating which was uniform, adherent, and free of cracks and porosities with considerably high hardness and wear resistant .The average surface roughness of the coating was found to be 13.5 mm and the coefficient of friction computed to be approximately 0.64 [33].

Laser alloying of AISI 1045 steel with TiC powder fed by the dynamic blowing method was carried out. By changing the laser power, scan speed and feed rate values, the depositions properties were studied. Optimum parameters significantly increased the surface hardness, and some dissolution of TiC in the molten Fe produced a small fraction of TiC dendrites upon re-solidification of the coating [34].

The wear resistance of tool steel against Sic abrasive grits was enhanced by a factor of up to about 6 by embedding TiC particles to a volume fraction of 50% and the experimental results showed that both the size of the reinforcing Tic particles and the structure of the matrix influenced the wear resistance [35].

The hardness increased by 30% by laser glazing and alloying of micro- and nano-particles of TiC on H-13 steel and a pronounced increase in its surface finish and increase in corrosion resistance was observed when a 1.5 kW CO_2 laser was used for its treatment [36].

The hardness of laser treated coating increased as Cu content and volume fraction of TiC particle on the Al-Mg alloy (A5083) as the substrate was increased and reached a maximum of HV500 without the cracking. The wear resistance of MMC layer increased as increasing hardness and reached approximately six times than that of the Al-Mg alloy substrate and also higher than that of the only Cu alloyed layer [37].

CHAPTER-3

PROBLEM IDENTIFICATION AND OBJECTIVE

- Problem identification and objective of the present work

Problem definition and objective of the present work

From the literature review it has been found that TiC coating by laser surface engineering improves the surface properties like hardness and wear resistance of various engineering materials. However, very little work related to TiC coating on AISI 304 steel have been done by laser surface coating technology to improve its surface properties. In the present work TiC coating has been developed on AISI 304 steel by Pulsed Nd: YAG laser and effect of different process parameters have been studied.

Objective of the present work are as follows

- To develop laser treated surface using TiC as coating material on AISI 304 stainless steel substrate such that the surface mechanical property of stainless steel is improved.
- To study the microstructure of laser clad cross section obtained by changing the laser processing parameters.
- To study the different phases obtained in the laser clad so formed by TiC on stainless steel.
- To find the hardness values of the laser clad so formed and compare it with constituent stainless steel.
- To study the effect of various laser processing parameters i.e. peak power, pulsed frequency and scan speed on hardness value and micro structure of the developed TiC coating.

CHAPTER-4

EXPERIMENTAL PLANNING AND PROCEDURE

- Materials and equipments used
- Experimental procedure
- Characterization of laser treated samples

4.1 Materials and Equipments used

The following materials and equipments are used for developing the laser coating and studying the microstructure and other properties of laser coating so obtained.

- Substrate-AISI304 stainless steel (25mmx50mmx5mm)

AISI304 stainless steel

Stainless steel types 1.4301 are also known as grades 304. Type 304 whose composition is mentioned in table 3 is the most versatile and widely used stainless steel. It is still sometimes referred to by its old name 18/8 which is derived from the nominal composition of type 304 being 18% chromium and 8% nickel.

AISI 304 steel has excellent corrosion resistance in a wide variety of environments and when in contact with different corrosive media. AISI 304 steel has good resistance to oxidation. AISI304 stainless steel cannot be hardened by heat treatment. Solution treatment or annealing can be done by rapid cooling after heating to 1010-1120°C. The typical physical properties of AISI304 stainless steel is shown in table 4.

AISI304 stainless steel is typically used in:

- Sinks and splash backs
- Saucepans
- Cutlery and flatware
- Architectural panelling
- Sanitary ware and troughs
- Tubing
- Brewery, dairy, food and pharmaceutical production equipment
- Springs, nuts, bolts and screws
- Medical implants

Table 3: Stainless steel composition (AISI304)

Fe	C	Si	Mn	P	S	Ni	Cr	Mo
Balance	0.067	0.753	1.731	0.045	0.031	8.554	18.97	0.224

Table 4: Typical Physical Properties of AISI 304 stainless steel

Property	Value
Density	8.00 g/cm^3
Melting Point	1400-1450°C
Modulus of Elasticity	193 GPa
Thermal Conductivity	16.2 W/m.K at 100°C
Thermal Expansion	17.2x10-6 /K at 100°C
Tensile strength (MPa)	520
Compression Strength (MPa)	210
Hardness Vickers(HV)	129

- Coating being used- TiC powder (size-325 mesh or 44µ)

Titanium carbide

TiC is an extremely hard refractory ceramic material whose physical properties are mentioned in table 5. It has the appearance of black powder with face cubic crystal structure. It is commercially used in tool bits. It is mainly used in preparation of cermets, which are frequently used to machine steel materials at high cutting speed.

Table 5: .Physical properties of TiC

Density (gm/cc)	Melting point (^{O}C)	Modulus of elasticity (GPa)	Coefficient of thermal expansion
4.93	3067	439	8.15-9.45x10^{-6} (25-1500)

- **Laser system used for present experiments**

Laser treatment of AISI304 steel with TiC powder as coating is done by using a 200W (maximum average power) pulsed Nd-YAG laser system [ALPHA LASER, GERMANY/ALT-200].

Detail specifications of the Nd-YAG laser system shown in fig 7 that has been used is as follows:

- Maximum average power=200W
- Pulse energy=90J
- Peak pulse power=10kW
- Pulse duration=0.5-20 ms
- Frequency=20Hz
- Focused diameter=0.2-2 mm
- 3- axis CNC Table

Fig .7: Pulsed Nd-YAG laser system

The process parameters that were considered during experiment were peak power (P_{peak}) (kW), the time for which substrate is exposed to laser per pulse (T_{on}) (ms), Frequency of pulsed laser (Freq) (Hz).

The average power (P_{avg}) was calculated considering the three parameters (P_{peak}, T_{on} and Freq) as:

$$P_{avg} = P_{peak} \times T_{on} \times Freq$$

- **Scanning electron microscope**

Scanning electron microscopy (SEM) uses a focused electron probe to extract external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample

The high spatial resolution of an SEM makes it a powerful tool to characterise a wide range of specimens at the nanometre to micrometre length scales. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties.

- **X-Ray diffraction**

X-ray diffraction (XRD) is an instrumental technique that is usually used to study crystalline materials. The three-dimensional structure of non-amorphous materials is defined by regular, repeating planes of atoms that form a crystal lattice. When an X-ray beam hits a sample and is diffracted, we can measure the distances between the planes of the atoms that constitute the sample by applying Bragg's Law.

Bragg's Law is $n\lambda=2d\sin\theta$, where the integer *n* is the order of the diffracted beam, 'λ' is the wavelength of the incident X-ray beam, 'd' is the distance between adjacent planes of atoms (the *d*-spacing), and 'θ' is the angle of incidence of the X-ray beam. These diffracted X-rays are then detected, processed and counted.

By scanning the sample through a range of 2θangles, all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacing allows identification of the mineral because each mineral has a set of unique d-spacing.

- **Microhardness testing machine (Vickers microhardness tester) :**

Micro indentation hardness testing (or microhardness testing) is a method for measuring the hardness of a material on a microscopic scale. A precision diamond indenter is impressed into the material at loads from a few grams to 1 kilogram. The impression length, measured microscopically, and the test load are used to calculate a hardness value. The hardness values obtained are useful indicators of a material's properties and expected service behaviour. Figure 8 shows the microhardness testing machine used for the measurement of hardness values.

Fig .8: Microhardness testing machine

4.2 Experimental procedure

Experiment was conducted in two phases. The initial phase was the preliminary experiment with substrate as mild steel and the second phase was the final experiment done with AISI304 stainless steel.

Preliminary experiment

Initially to determine the range of peak power and frequency and scan speed that has to be considered during the laser coating of stainless steel, experiment was done on mild steel sample and coating powder was taken to be TiC. During the experiment the laser treatment was done by taking different peak powers (1-3 kW), frequencies (5-18 Hz), T_{on} time (6-12 ms), and scan speed (2.5-10 mm/s) which is tabulated in table 6. The spot diameter was kept constant and was taken as 1.5 mm.

Table 6: .Experimental parameters and their values taken to develop laser coating of TiC on mild steel

Sample No.	Sl. No.	Spot diameter (mm)	Speed mm/s	Frequency (Hz)	Ton (ms)	Peak power (kW)
Sample 1						
	1	1.5	10	15	12	1
	2	1.5	10	15	12	2
	3	1.5	10	10	12	1
	4	1.5	8	15	12	1
	5	1.5	2.5	15	12	1
	6	1.5	6	15	12	1
	7	1.5	12	15	12	0.3
	8	1.5	12	15	12	1
	9	1.5	10	15	12	0.7
	10	1.5	10	15	12	0.5
Sample 2						
	1	1.5	10	15	12	1.2
	2	1.5	10	12	12	1.4
	3	1.5	10	9.5	12	1.8
	4	1.5	10	15	12	1
	5	1.5	2.5	5.5	12	3
	6	1.5	2.5	4.2	12	4
	7	1.5	5	5.5	12	3
	8	1.5	10	5.5	12	3

After the above experiment was conducted, it was found that in experiments 7-10 of sample 1 which were laser treated with peak-powers less than or equal to1kW and high scan speeds of 10-12mm/s, coating layer of TiC was formed but this particular layer did not form bond with steel substrate. Thus such low powers are unsuitable for forming high bond strength coatings which are used in cutting tools.

Also it was observed that in experiments 5-8 of sample 2 with peak power in the range 3-4kW and scan speed of 2.5-5 mm/s the coating surface so formed was non uniform and rough. This is because of spattering of TiC by laser due to low speed and high power, therefore high peak power and low scan speed is not suitable for coating of TiC using laser due high penetration effects.

Thus we can see that better coating surfaces are formed in the power range of 1-2 kW and frequency of 15Hz and at a lower scan speed.

Figure 9 shows the mild steel sample treated with laser using TiC as coating powder with different parameter combination.

Fig .9: Laser coating on mild steel with different parameter combination

Final experiment

After the preliminary experiment the range of peak power, frequency and scan speed suitable for optimum coating of TiC on stainless steel was obtained and was worked upon in the final experiment.

Preparation of substrate for laser coating

Firstly the AISI304 substrate of dimensions 25mmx50mmx5mm was polished on the faces to obtain required surface finish. Then the substrate was cleaned with alcohol and acetone respectively to remove any unwanted particles, dust or burr. Coating powder which was TiC in this experiment is mixed with a binder. The binder used in this case was dendrite which is a semi solid, was liquefied by using acetone. The solution of TiC, acetone and dendrite was constantly stirred with the help of a glass rod until the solution becomes homogeneous. The homogeneous solution so formed was spread over the substrate to get a uniform thickness of the coat layer. The green coating was allowed to dry in normal atmospheric condition. Figure 10 and fig 11 show the step by step preparation of substrate sample for the operation of laser treatment.

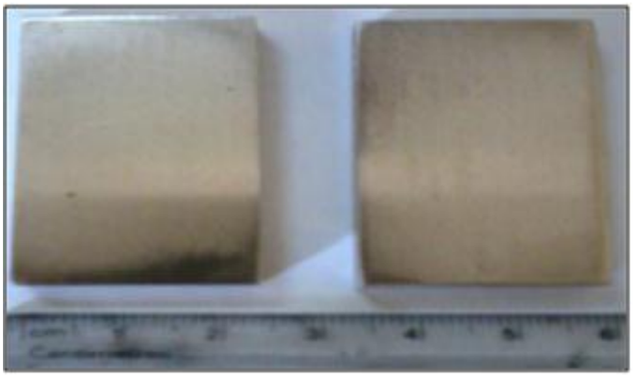


Fig.10: Stainless steel samples before TiC powder coating

Fig .11: Green coating of TiC powder over stainless steel substrate

Flow chart for preparation of sample prior to laser treatment:

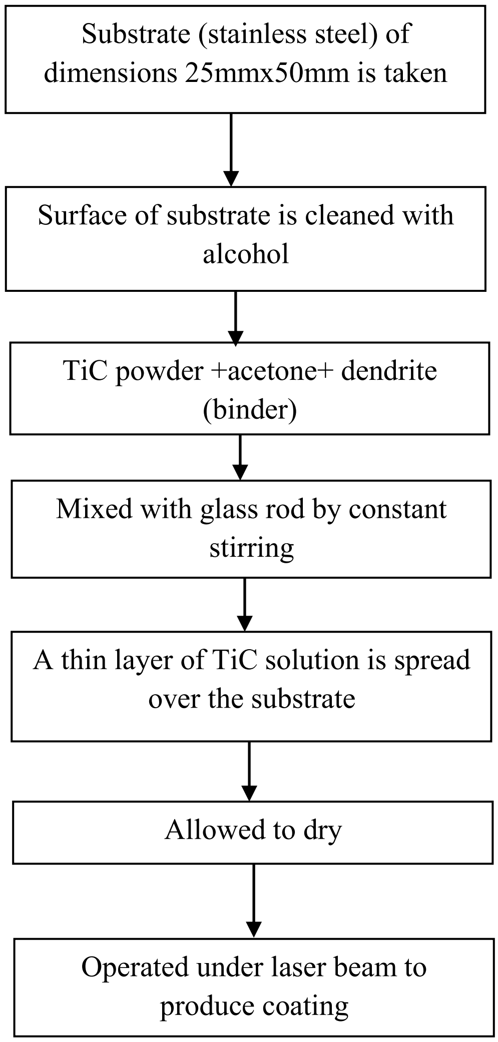
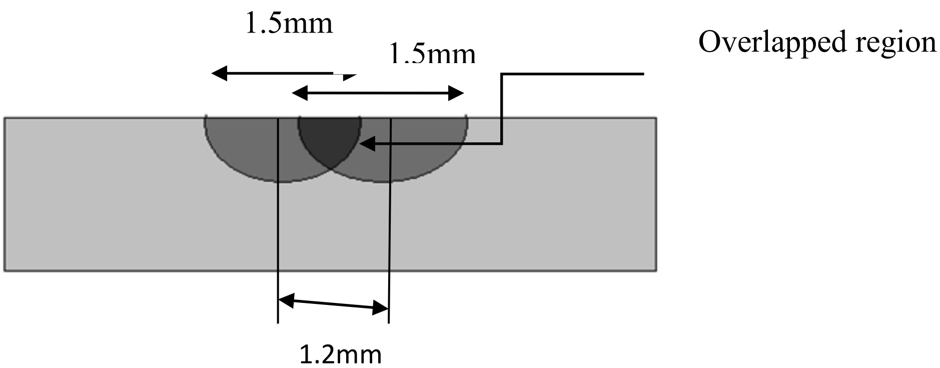


Fig .12: Flow chart for the preparation of substrate

Laser treatment

To form a wider coating area with laser beam of spot diameter of 1.5 mm, overlapping between two subsequent scans was done as shown in fig 13(b). Distance between two parallel scan was taken 1.2 mm. The no of overlapped scans done in a particular condition of parameters was 5 as shown in fig 13(a). Distance between two tracks of different condition was taken 4 mm as depicted in fig 13(a). Figure 14 shows the laser treated sample of stainless steel.

(a)



(b)

Fig .13: Dimensions of tracks and each scan (a) top view of sample (b) side view of sample

Fig .14: Laser treated coating tracks of TiC on stainless steel

Total 16 numbers of experiments have been conducted in different combination of laser process parameters. Details of the experimental condition are given in Table-7.

Table 7: Process parameters during laser coating experiment on stainless steel

Exp. No.	Sample No.	Track No.	Peak power (kW)	T_{on} (ms)	Frequency (Hz)	Scan speed (mm/s)
1	1	1	1	9	15	4
2		2	1	9	15	6
3		3	1	9	15	8
4		4	1	9	15	10
5	2	1	1	9	18	4
6		2	1	9	18	6
7		3	1	9	18	8
8		4	1	9	18	10
9	3	1	1.2	9	15	4
10		2	1.2	9	15	6
11		3	1.2	9	15	8
12		4	1.2	9	15	10
13	4	1	1.2	9	18	4
14		2	1.2	9	18	6
15		3	1.2	9	18	8
16		4	1.2	9	18	10

4.3 Characterization of laser treated samples

SEM analysis

SEM is used to study the detailed microstructure of the laser treated TiC coating on AISI 304 stainless steel. For this the samples was cut across the length perpendicular to the laser scan direction in such a way that the cross section of the laser clad was exposed. The cross section area was polished with coarse emery paper to remove the rough area obtained after cutting of the sample. Then it was polished with fine emery papers of grades 1, 2, 3 and 4(in the increasing order of fineness) respectively. Finally the sample was cleaned with acetone before testing it under SEM. The microstructure of the coating had been studied under SEM (JEOL-648OLV). The micrographs have been taken in the BSE (back scattered electron emission) mode.

XRD analysis

The different phases of the laser clad were obtained by X'Pert (company-PAN analytical, product no-3040/00) X-ray Diffractometer with CuKα (λ= 1.5418 Å) radiation. The scanning range was taken 20^0-100^0. The step size under each study was taken 3^0/min. The XRD patterns were analysed with the help of Phillip's X'Pert High Score software.

Micro hardness analysis

Microhardness of the developed coating was measured under Leco microhardness tester (company -LM248AT) with 100gf indenting force and dwell time of 10s. Average hardness of each track having individual set of laser processing parameters, was found by taking 8-10 hardness measurement.

CHAPTER-5

RESULTS AND DISCUSSIONS

- Phase analysis by XRD
- Microstructure analysis under SEM
- Micro-hardness measurement and effect of process parameters on hardness

5.1 Phase analysis by XRD

The phase analysis by XRD gives the following plots (Fig 15 and Fig 16) of intensity of elements vs angle of incidence rays (θ) under different laser processing conditions.

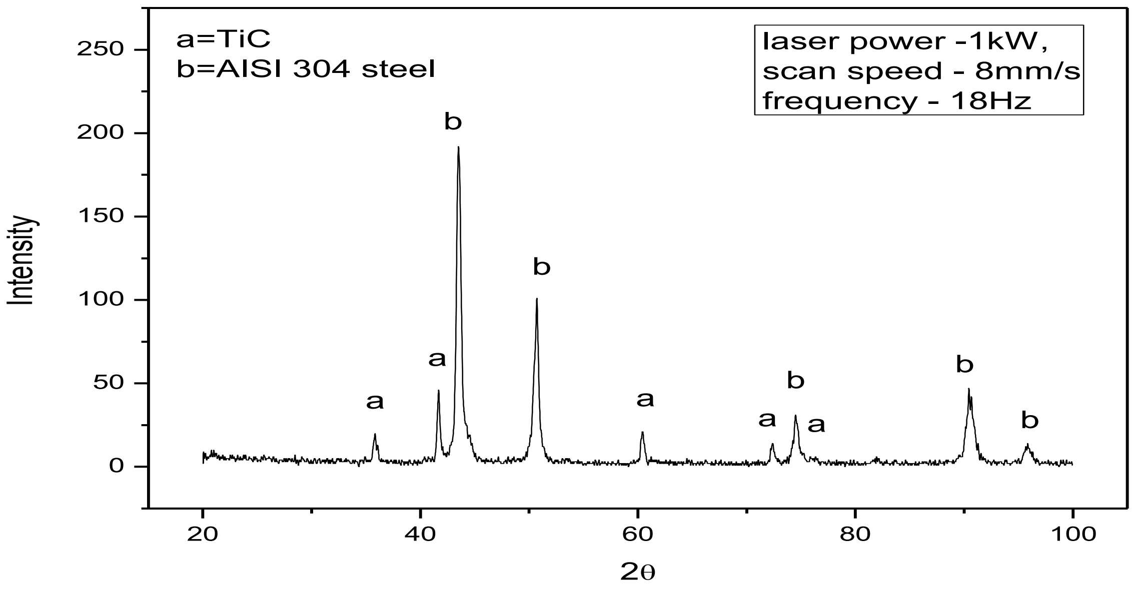


Fig .15: XRD spectrum of the surface of AISI304 stainless steel laser coated with TiC when processed with peak power of 1kW, scan speed of 8mm/s and frequency of 18Hz

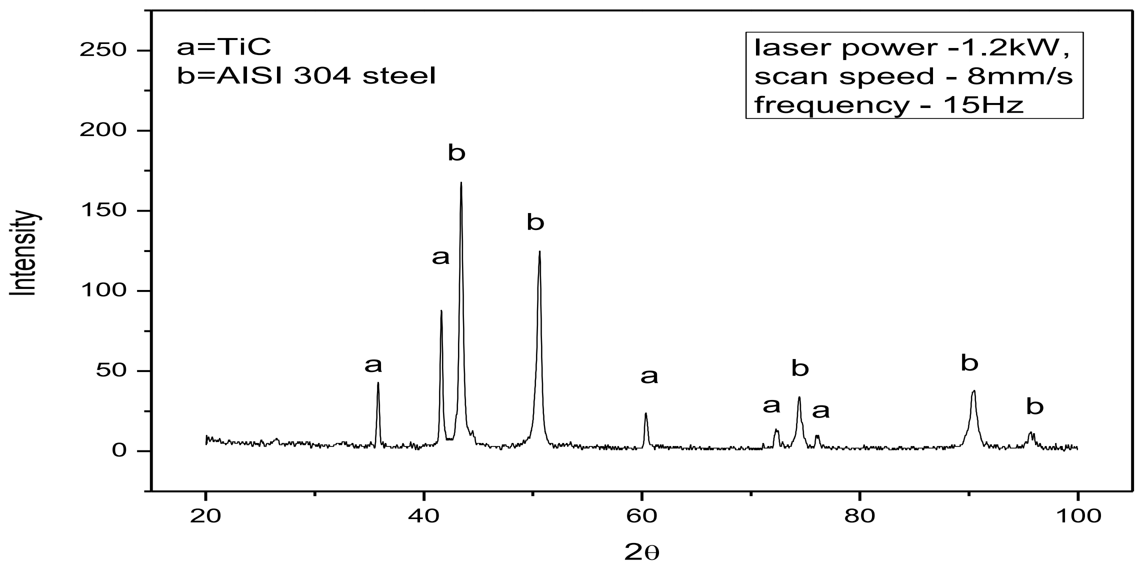


Fig .16: XRD spectrum of the surface of AISI304 stainless steel laser coated with TiC when processed with peak power of 1.2 kW, scan speed of 8mm/s and frequency of 15Hz

The XRD spectrum obtained in fig.15 and fig.16 are of samples having two different conditions. The figures show the constituent phases of laser coating so obtained were that of TiC and Fe which was same as that of coating powder and the substrate. Thus it clearly indicates that there is no reaction between TiC and Fe but they occur in free states in the laser processed zone. Thus in both the conditions constituent phases remain the same. The results show that in fig.15 the intensity of TiC as compared to fig .16 is less, thus the amount of TiC deposited in sample with parameters peak power of 1kW, scan speed of 8mm/s and frequency of is more than that of sample with parameters peak power of 1.2 kW, scan speed of 8mm/s and frequency of 15Hz. We can see that with increasing frequency the intensity of TiC phase decreases keeping the scan speed constant.

Also we see that by increasing the peak power, the intensity of Fe is more indicating that the increase in power increases the penetration of TiC into the substrate and higher exposure of stainless steel on the surface.

We can also see that changing the parameters only changed the relative amounts of TiC and Fe phases but did not change the phase constituent of the clad.

5.2 Microstructure analysis under SEM

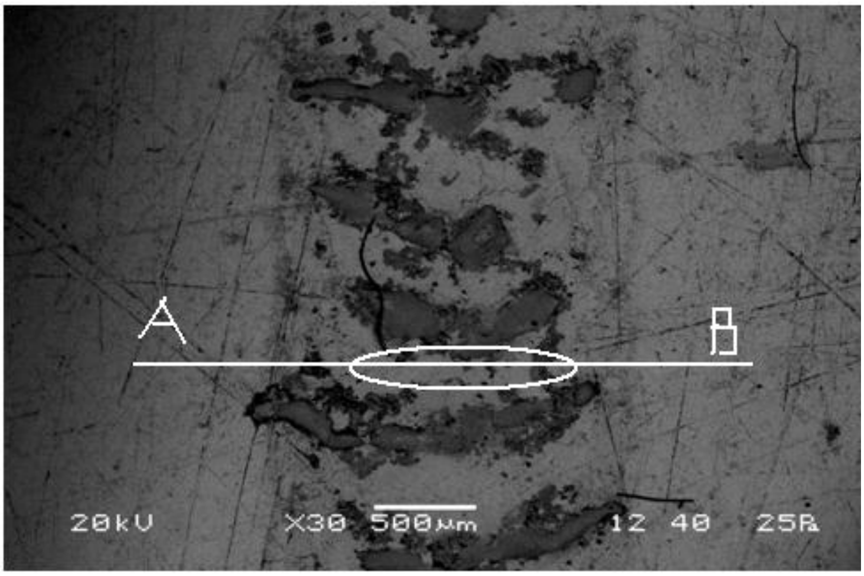


Fig .17: Top view of the laser coated track prepared with peak power of 1.2kW, scan speed of 6mm/s, Ton time of 9ms and frequency of 18Hz

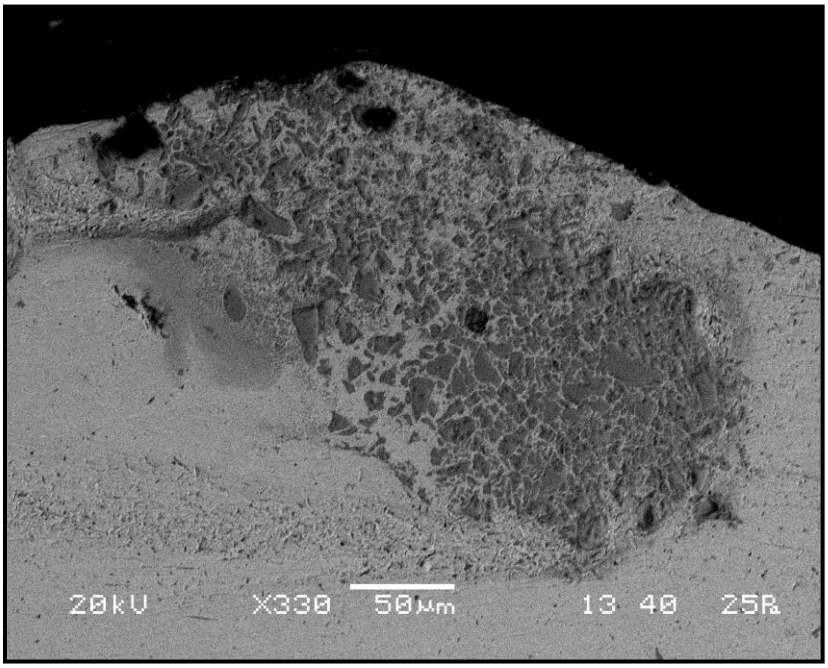


Fig .18: The magnified SEM image of the cross section of the laser coated stainless steel processed with peak power of 1.2kW, scan speed of 6mm/s, Ton time of 9ms and frequency of 18Hz

Figure 17 shows the top view of laser treatment done on stainless steel substrate by using TiC as coating powder. In the SEM images the dark region shows the TiC and the grey region is the stainless steel. From the figure we can see that TiC has been spattered at larger areas keeping majority area of the track uncovered. The reason behind such a profile is high peak power of 1.2 kW and high frequency of 18 Hz. High values of peak power and frequency increase the penetration depth (both are directly proportional to penetration) and thus dispersing TiC at the circumference of the laser spot and exposing the substrate.

Figure 18 shows the SEM image of the encircled region when the sample was cut at section A-B. The magnified image of the cross section of the encircled region which shows a uniformly dispersed MMC type coating of TiC on steel matrix formed at major portions of the track as shown in fig 18.

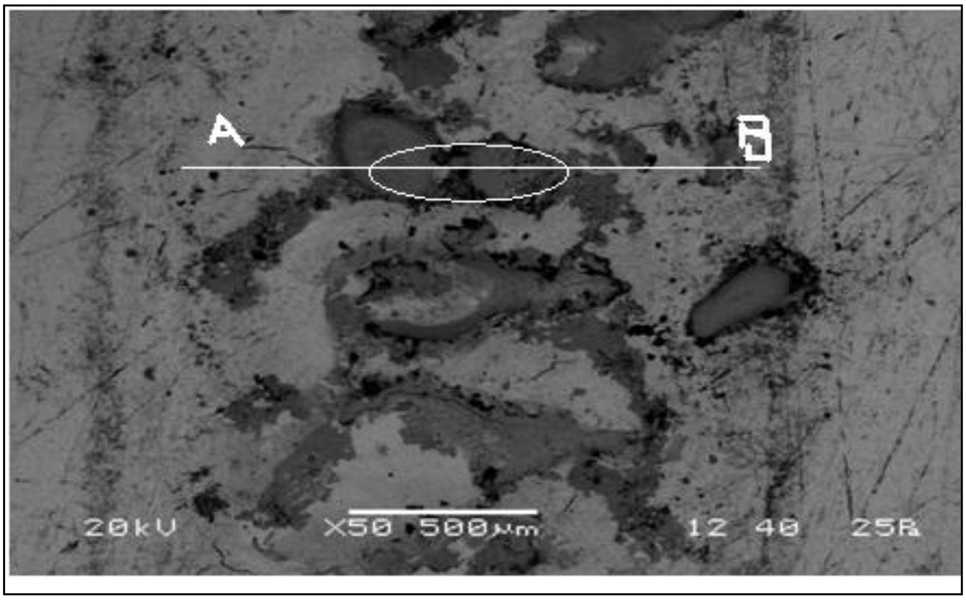


Fig.19: Top view of the laser coated track prepared with peak power of 1kW, scan speed of 8mm/s, Ton time of 9ms and frequency of 15Hz

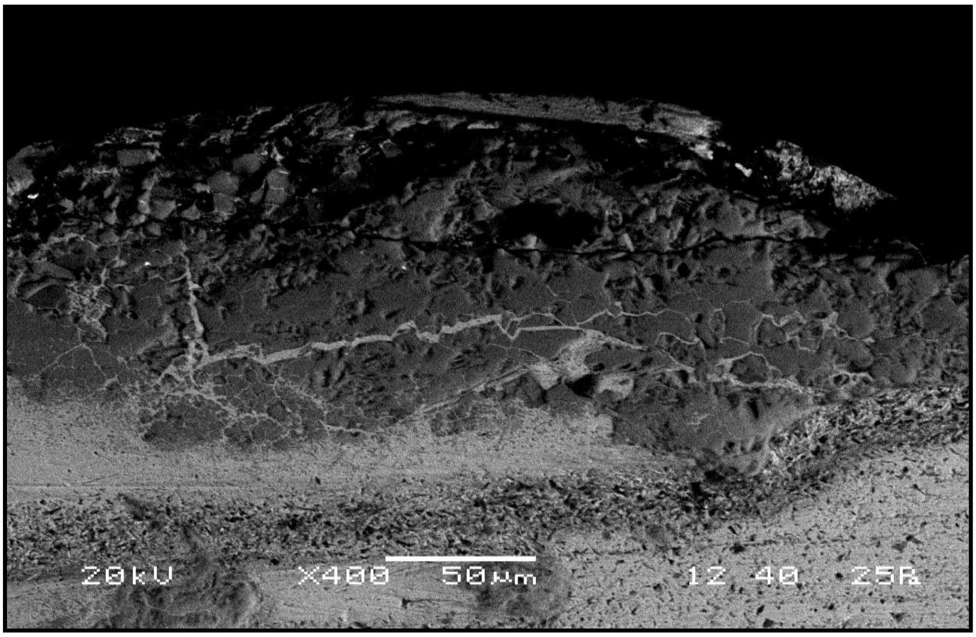


Fig. 20: The magnified SEM image of the cross section of the laser coated stainless steel processed with peak power of 1kW, scan speed of 8mm/s, Ton time of 9ms and frequency of 15Hz

Figure 19 shows the top view of laser treatment done on stainless steel substrate by using TiC as coating powder with peak power of 1kW, scan speed of 8mm/s, Ton time of 9ms and frequency of 15Hz. In this figure we can see that TiC coating has been deposited on greater amount of areas as compared to fig 17 and the patterns obtained in fig 19 are distorted. This is because the track was obtained taking processing parameters as peak power 1kW and frequency 15 Hz which were comparatively less as compared to that taken in fig 17 thus decreasing the penetration depth.

Figure 20 shows the magnified SEM image of the cross section at encircled region obtained by cutting the sample at section A-B where maximum amount of TiC has been deposited on the substrate surface.

Thus we can say that the type of coatings and the coating thickness obtained vary with changes in laser processing parameters.

5.3 Micro-hardness measurement and effect of process parameters on hardness

5.3.1. Effect of laser scan speed on hardness

To find the effect of laser scan speed on microhardness of the laser coating obtained, a plot between microhardness measured in HV (Vickers hardness) (load taken 100gm) vs laser scan speed measured in mm/s was drawn.

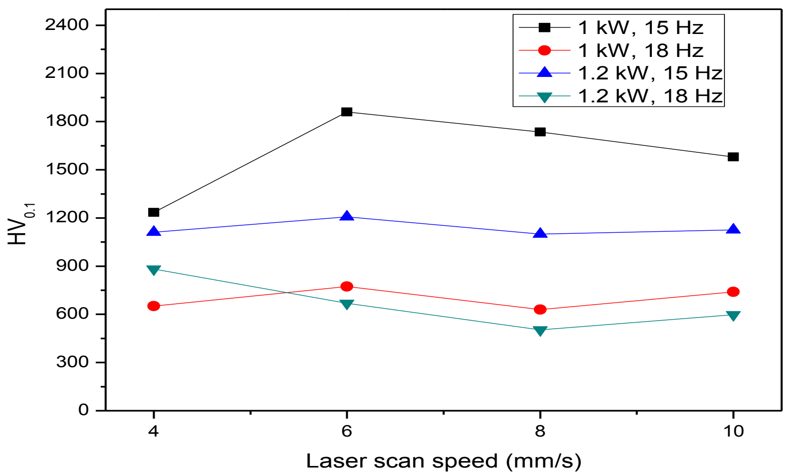


Fig .21: Variations of micro hardness value against laser scan speed

The above plot (Fig 21) shows the values of hardness at different laser processing parameters, each curve with distinctive colour represents a particular peak power and frequency. From the plot it can be seen that no particular pattern of hardness values is being followed with changing scan speed. Thus we can conclude that effect of laser scan speed in case of coating obtained by pulsed laser is unpredictable.

5.3.2. Effect of laser pulse frequency on hardness

To find the effect of frequency on microhardness of the laser coating obtained, plot between microhardness measured in HV (with load 100gm) vs laser pulse frequency measured in Hz was drawn (Fig.22 and Fig.23).

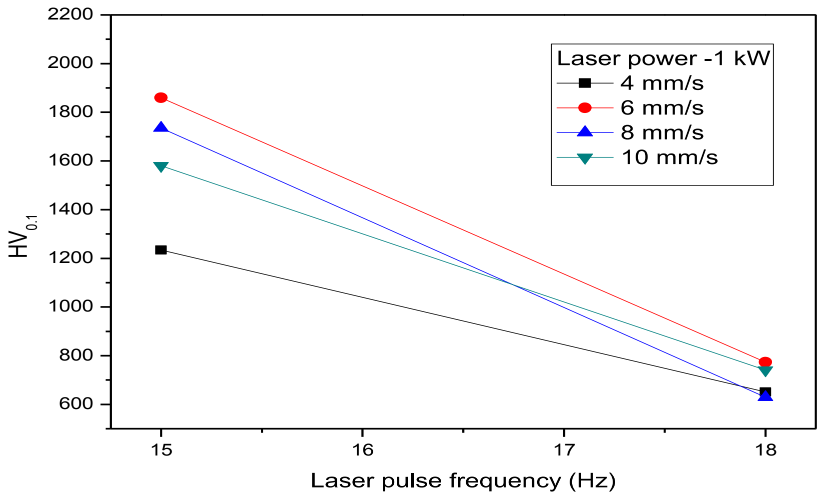


Fig .22: Variations of micro hardness value against laser pulse frequency for laser power 1 kW

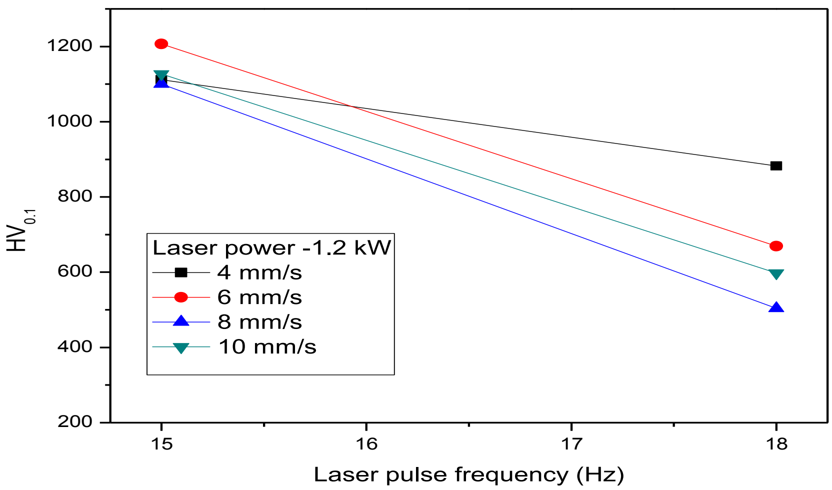


Fig .23: Variations of micro hardness value against laser pulse frequency for laser power 1.2 kW

The plot (Fig 22) has been obtained by taking hardness values keeping peak power as 1kW and scan speed constant (4,6,8 and 10 mm/s respectively) for each linear curve at two different frequencies of 15Hz and 18Hz. It can be seen that hardness value decreases with increasing frequency for each curve. This is because with increasing frequency the laser penetration into the substrate increases thus exposing more of substrate at surface. And since substrate being stainless steel has lesser hardness than the TiC, the hardness value on the curve drops.

Figure 23 has been obtained by taking hardness values at two different frequencies of 15Hz and 18Hz at different scan speeds (4, 6, 8 and 10 mm/s respectively) keeping peak power constant as 1.2 kW for each linear curve. It can be seen that hardness value decreases with increasing frequency for each curve justifying similar explanation as in fig 22.

5.3.3. Effect of laser power on hardness

To find the effect of laser peak power on microhardness of the laser coating obtained, plot between microhardness measured in HV (with load 100gm) vs peak power measured in kW was drawn (Fig. 24 and Fig.25).

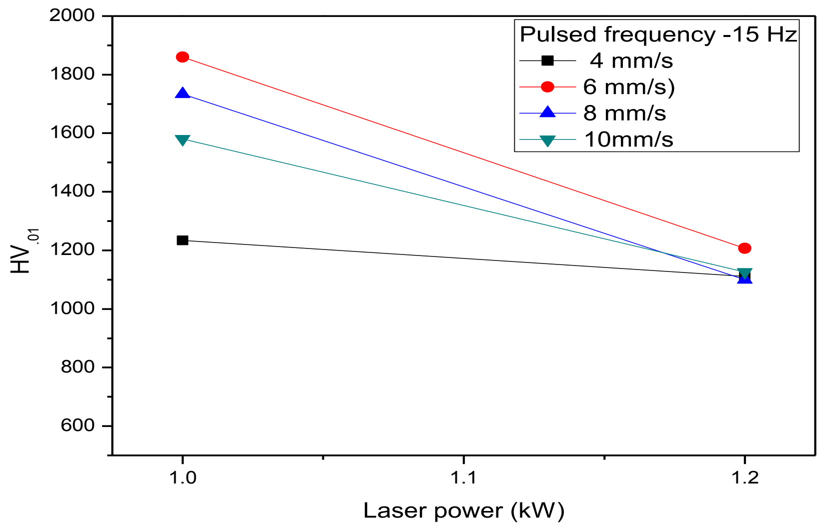


Fig .24: Variations of micro hardness value against laser power for laser pulse frequency 15 Hz

The plot (Fig 24) has been obtained by taking hardness values at laser powers 1kW and 1.2 kW respectively for different scan speeds (4, 6, 8 and 10 mm/s respectively) at constant frequency of 15Hz. We can see that with increasing peak power at particular scan speed and frequency, hardness values decreases. This is because with increasing power the depth of penetration increases, exposing more of substrate at surface which has lesser hardness than the coating TiC.

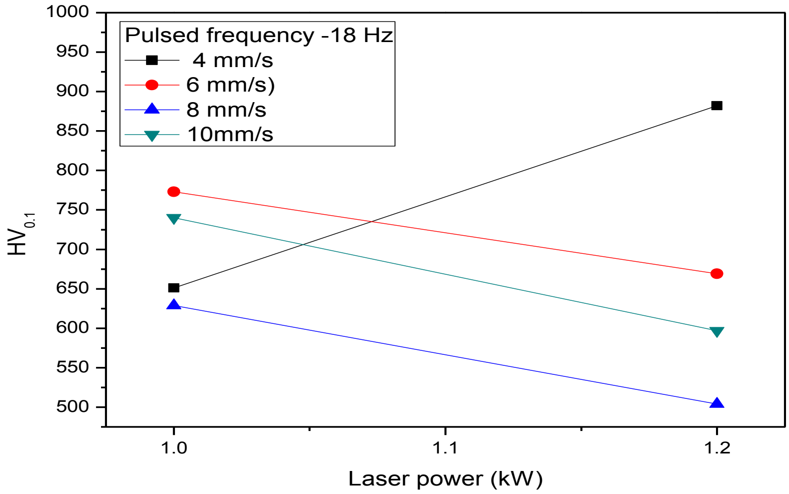


Fig .25: Variations of micro hardness value against laser power for laser pulse frequency 18 Hz

Figure 25 where a plot of microhardness vs laser power is drawn keeping frequency as 18Hz shows similar trend of hardness values as shown in fig 24 where frequency was taken 15Hz. But noticeable thing is that the corresponding hardness values at 1kW and 1.2kW are less in fig 25 than in fig 24. This is because of the fact that more of substrate comes to the surface with increasing frequency. Also an abrupt change in the trend of hardness value was obtained in case of scan speed of 4mm/s. This is very difficult to explain because this could be probably a result of uncertainty in the coating uniformity.

CHAPTER-6

CONCLUSIONS AND FUTURE SCOPE

- Conclusions
- Future scope

6.1 Conclusions

- From the above experiment we can conclude that TiC coating has been successfully developed on AISI 304 steel.

- Laser treatment by using TiC as coating material, increases the surface properties of the stainless steel like its hardness without actually affecting its bulk mechanical properties.

- We can also conclude that the surface properties are highly affected by the laser process parameters.

- From the XRD analysis we can say that by increasing the peak power (from 1 kW to 1.2 kW) and laser pulse frequency (15Hz to18Hz) the intensity of TiC phase on the surface decreases and the intensity of Fe increases keeping the scan speed constant.

- From the microstructure analysis we can conclude that at low values of frequency and peak power a thick coating of TiC is more uniformly distributed over the laser treated tracks. And at higher values of frequency and peak powers a MMC type of coating is obtained in which TiC is dispersed over the substrate.

- It was found that hardness value of the laser treated surface increased to values in the range of 650-1900 $HV_{0.1}$ depending on laser processing parameters.

- When effect of laser scan speed on hardness value was studied no peculiar relation has been observed.

- From the experimental results it has been observed that Hardness value of the developed TiC coating is decreases with increasing laser pulse frequency when peak power is constant.

- It has also been observed that Hardness value of the developed TiC coating, decreases with increasing value of laser peak power keeping frequency constant.

6.2 Future scope

- Study the tribological behaviour (wear resistance, coefficient of friction) of the developed TiC coating.

- Development of in-situ laser cladding process for exhibiting benefits of production of coating powder within the laser system itself and using TiC as reinforcement and along with it some other metals which serve as matrix and improve the surface properties further.

- Use of continuous working high power diode lasers, fibre lasers and sophisticated knowledge based controllers that help in producing uniform laser coating which is less susceptible to variations in process parameters.

CHAPTER-6

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